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A study of compressive strength between zirconia framework and veneering ceramic as a function of thermal expansion coefficient using Shell Nielsen test method

Bulent Gokce, DDS, PhD,^a / Hamit Serdar Cotert, DDS, PhD^b / Mutlu Özcan, Dr.med.dent., PhD^c

^a*Associate Professor, Department of Prosthodontics, Ege University, School of Dentistry, Izmir, Turkey*

^b*Professor, Department of Prosthodontics, Ege University, School of Dentistry, Izmir, Turkey*

^c*Professor, University of Zurich, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland*

Short title: *Bond strength of veneering ceramics to zirconia*

Correspondance to: *Dr. Bülent Gökçe, Ege University, School of Dentistry, Department of Prosthodontics, 35100 Bornova, Izmir, Turkey, Tel: +90-533-4119869; Fax: +90-232-2880325. e-mail: bulentgokce@yahoo.com*

Abstract: This study investigated the bond strength between a zirconia framework and four veneering ceramic materials with varying coefficients of thermal expansions (CTE) using the Shell Nielsen bond strength test. Zirconia rods (N=40) (ICE Zirkon) (diameter: 4 mm, height: 20 mm) were milled in green state and sintered. After firing, the zirconia rods were air-abraded, ultrasonically and steam cleaned. They were randomly assigned to receive four veneering ceramic materials (n=10/group), namely a) Vita VM9 (VZ; $9-9.2 \times 10^{-6} \text{K}^{-1}$), b) Cerabien ZR (CZ; $9.1 \times 10^{-6} \text{K}^{-1}$), c) Matchmaker ZR (MM; $9.4 \times 10^{-6} \text{K}^{-1}$) and d) Ice Zirconia Ceramic (IZ; $9.6 \times 10^{-6} \text{K}^{-1}$). The veneering ceramics were then fired onto zirconia discs (height: 2 mm, thickness: 2 mm) circumferentially and were thermocycled for 6.000 times (5/55°C, dwell time: 30 s). Specimens were loaded from the top of the zirconia rods (crosshead speed: 0.5 mm/min) in a universal testing machine until debonding. Shell Nielsen bond strength values were calculated and expressed in MPa. The surfaces of the zirconia rods were evaluated under Scanning Electron Microscopy to determine the failure types. The data were then statistically analyzed using 1-way ANOVA and Tukey's tests ($\alpha=0.05$). Weibull distribution values including the Weibull modulus (m), characteristic strength (σ_0), and probability of failure at 5% (0.05) was calculated. The highest mean bond strength (MPa) was obtained for CZ (42.08 ± 4.08), followed by VZ (41.77 ± 4.92), MM (40.7 ± 3.64) and IZ (40.05 ± 5.78). While mean bond strength for VZ, MM and IZ were not significantly different ($P > 0.05$), CZ was only significantly higher than that of IZ ($P < 0.05$). The Weibull distribution presented the lowest shape value (m) for VZ ($m=16.94$) and the highest for MM ($m=20.16$). The specimens in all test groups demonstrated predominantly adhesive failure type between the framework and veneering ceramic followed by mixed failures. Veneering ceramics with a greater mismatch of CTE with the zirconia framework exhibited similar Shell Nielsen bond strength to those with fewer mismatches. Shell Nielsen bond strength test used in this study subjecting the zirconia framework-veneering ceramic assemblies to circumferential shear forces did not show the effect of CTE mismatch between the veneering ceramics except those between CZ ($9.1 \times 10^{-6} \text{K}^{-1}$) and IZ ($9.6 \times 10^{-6} \text{K}^{-1}$).

Keywords: Adhesion, bond strength, chipping, veneering ceramic, zirconia

Introduction

Zirconium dioxide (ZrO_2 , hereon: Zirconia) has become an alternative to metals as a framework material for veneered anterior and posterior fixed dental prosthesis (FDP) due to its relatively superior mechanical, chemical, biological and optical properties [1-3]. Although monolithic zirconia FDPs have become popular in recent years, in most cases zirconia frameworks are veneered to enhance the optical properties of the final restoration. The mechanical strength, stability of the framework, and the adhesion of the veneering ceramic (VC) to the framework are crucial factors for clinically successful performance and reliability of a veneered zirconia FDP [4,5].

Mechanical properties of framework materials and VCs should be consistent to achieve a durable bond [3,4]. Bond strength of a VC to zirconia is affected by many variables such as restoration geometry, framework design, surface finish, roughness and treatment, wettability of zirconia framework, phase transformation, structural defects yielding to flaw development, reactions at the framework-veneer interface, residual tensile stresses during the cooling process and Coefficient of Thermal Expansion (CTE) mismatch between framework and the VC [4-7]. Individual or the combined effects of all these variables could influence the framework-veneer bond strength and therefore the clinical success rate of FDPs made of such materials [4-7].

Clinical and laboratory investigations demonstrated that zirconia frameworks have a low failure rate compared to VCs [8,9]. Clinical failures such as chipping and/or delamination of veneered zirconia in all major brands were reported to be between 11.4% and 25% mainly in posterior FDPs [10,11]. These failures were attributed to poor bond strength at the framework-veneer interface or cohesive strength of the VC [2,7,12-14].

In bilayered structures such as a veneered FDPs, stress distribution is much more complex compared to a monolithic structure [2]. The inherent characteristics of the framework, VC and the bonding interface between these two materials causes the complexity [15,16]. For better clinical performance, it has been proposed to use specially developed VCs for both layering and press techniques and to treat the surface of the zirconia to enhance the adhesion at the framework-veneer interface. Airborne particle abrasion to roughen and clean the zirconia surface for higher bond strength between these two ceramics is being routinely used [17-19]. Application of a liner for the same purpose has also been suggested [17].

In order to understand and evaluate the bond strength and failure types of these systems various in-vitro test designs were developed. These tests were adopted to understand the mechanical nature of bilayered ceramics, find solutions for specific problems and predict in-vivo survivability [17]. Shear bond strength (SBS), macrotensile, microtensile bond strength (MTBS), biaxial, 3- and 4- point flexure strength tests have been suggested to investigate the framework-veneer bond strength in in-vitro conditions [12,13, 20,21]. These tests use standardized non-anatomical specimens under controlled environments and parameters and give valuable information about the basic mechanical properties of bilayered structures. However, they do not represent the complex geometry of the FDPs [22] as they are based on applying a tensile or a compressive load on one side of the veneered specimen [12,13,20,21]. In fact, in clinical conditions the framework is veneered circumferentially resulting in radial compressive forces that enhance the bond strength at the framework-veneer interface [23]. On the other hand, bending or flexural strength tests, favorable for metal-ceramic systems, may not be valid for veneered ceramic materials due to their brittle nature [24,25]. Commonly used SBS and MTBS tests often show cohesive failure patterns within the VC. Thus, the results do not represent the actual bond strength at the interface [5,26,27]. Each test setup plays an important role in determining the framework-veneer bond strength and has limitations in measuring the interfacial bond strength [1,2,6,7,12,24,28]. Anatomical restorations were also fabricated and loaded to closely mimic the clinical situation but the data obtained in these studies is more difficult to compare due to substantial variations in test parameters [29].

The test specimen design and methodology proposed by Shell and Nielsen [23] provided valuable information regarding the bond strength of VC to framework material. The design was based on the measurement of the load required to fail the circumferentially fired layering ceramic around the rod shaped substructure, measuring the shear bond strength at the core-veneer interface similar to the push-out test [23]. The Shell Nielsen [23] test methods and the Mc-Lean test [30] methods have been used to quantify the ultimate and actual bond strength at the framework-veneer interface [30].

The objectives of this study were to a) investigate the bond strength between one type of zirconia framework material and four feldspathic VCs with different CTEs using the Shell Nielsen bond strength test and b) determine the failure types after debonding using scanning electron microscopy. The null hypothesis tested was that the CTE differences between the framework and the VCs would not influence the bond strength.

Materials and Methods

Preparation of rod shaped zirconia framework specimens

The materials used in this study are listed in Tables 1 and 2.

The design of the specimens for testing the bond strength between VC and zirconia rods was based on the design described by Shell and Nielsen [23]. Zirconia rods (Ice Zirkon, ZirkonZahn GmbH, Bruneck, Germany) (N=40) were milled in green state then sintered (diameter: 4 mm, height: 20 mm). After sintering, the rods were airborne particle abraded (120 μm Al_2O_3 ; pressure 350 kPa) according to the manufacturer's conditioning recommendation. Zirconia rods were then ultrasonically and steam cleaned. Finally, veneering ceramics were fired onto the zirconia rods. Detailed firing schedules for each veneering ceramic is presented in Table 3.

Shell-Nielsen bond strength test

The stainless steel mould was placed on a plane surface and the veneered zirconia rods were seated back into the mould that had been used for specimen preparation (Fig 1). The specimens were loaded from the top of the zirconia rods with a ball point at a crosshead speed of 0.5 mm/min in the universal testing machine (Autograph Model AG-50kNG, Shimadzu, Kyoto, Japan) (Fig. 2) until fracture of the VC (Fig. 3). Shell-Nielsen bond strength values were calculated, expressed in MPa and stress-strain curves were analyzed.

Failure type analysis

Complementary to the bond strength tests, the surfaces of the zirconia rods were evaluated after the tests under the Scanning Electron Microscope (SEM) (JEOL JSM-5200, Kyoto, Japan). Failure types were categorized as follows: Adhesive (A): Failure between the framework and the veneer ceramic without any remnants of the veneer ceramic; Cohesive (C): Failure within the veneering ceramic; Mixed (M): Combination of A and B.

Statistical analysis

Descriptive statistics were computed and test of normality was performed using Kolmogorov-Smirnov and Shapiro-Wilk's tests (SPSS 14.0, Chicago, IL, USA). The means of each group were analyzed by 1-way analysis of variance (ANOVA), with Shell and Nielsen bond strength as the dependent variable and

veneering ceramics with different TEC (4 levels: MM, $9.4 \times 10^{-6} \text{ K}^{-1}$, CZ, $9.1 \times 10^{-6} \text{ K}^{-1}$, IZ, $9.6 \times 10^{-6} \text{ K}^{-1}$, VZ, $9.2 \times 10^{-6} \text{ K}^{-1}$). Tukey's post hoc test was applied to compare the significant differences between groups. Maximum likelihood estimation without a correction factor was used for 2-parameter Weibull distribution, including the Weibull modulus, scale (m) and shape (σ_0), to interpret predictability and reliability of adhesion (Minitab Software Version 14, State College, PA, USA):

$$\ln \ln \frac{1}{1-F(\sigma_c)} = m \ln \sigma_c - m \ln \sigma_0$$

$P < 0.05$ was considered to be statistically significant in all tests.

Results

Data were normally distributed in all groups. Mean bond strength, standard deviations at the framework-veneer ceramic interface, and failure types of the tested groups are summarized in Table 4.

The highest mean bond strength (MPa) was obtained for CZ (42.08 ± 4.08), followed by VZ (41.77 ± 4.92), MM (40.7 ± 3.64) and IZ (40.05 ± 5.78). While mean bond strength for VZ, MM and IZ were not significantly different ($P > 0.05$), CZ was only significantly higher than that of IZ ($P < 0.05$). VZ, MM and IZ were not significantly different ($P > 0.05$).

Weibull distribution presented the lowest shape value (m) for VZ ($m = 16.94$) and the highest for MM ($m = 20.16$) (Table 4, Fig. 4).

The specimens in all test groups demonstrated predominantly adhesive failure type (A) between the framework and veneering ceramic (IZ: 5, VZ: 7, MM: 6, CZ: 7) followed by mixed (M) failures (IZ: 4, VZ: 2, MM: 3, CZ: 3) (Figs. 5-c) (Figs. 5 d,e). Cohesive (C) failure within the veneering ceramic was the less common (IZ: 1, VZ: 1, MM: 1, CZ: 0) (Fig. 5f).

Discussion

Based on the results of this study, since the CTE differences between the VCs did not significantly affect the bond strength between the framework and the veneer ceramic except between groups, the null hypothesis tested could be partially accepted.

Mechanical laboratory tests, such as bond strength tests, guide clinicians concerning materials, procedures and methods [31]. In attempt to estimate the bond strength and failure characteristics of VCs to

zirconia, various laboratory tests (shear, microtensile, biaxial flexure and 3-, 4- point flexure strength tests) have been conducted [32-35]. There is a great variation between the results of these tests due to differences in testing methodology and thereby the fracture mechanism. Therefore, it is difficult to interpret the bond strength values gathered from various studies [12,13,20,21].

The MTBS test is advocated to evaluate the framework-veneer bond strength as a more standardized testing procedure with smaller standard deviation [7,36]. Higher rates of cohesive failures within the layering ceramic have been reported with the MTBS test and it gives less information about the ultimate bond strength between the framework and the VC [17,31]. Furthermore, in clinical and laboratory conditions, the incidence of delamination of the VC is more frequent than the cohesive failures within the VC [19,20,37,38]. Therefore, the MTBS test might be questionable as a means to evaluate the bond strength between the framework and the VC.

SBS tests provide valuable information about the materials tested. However, variations in test geometry and loading configuration have a high impact on the stresses that are generated at the framework-veneer interface with this test method [39]. In SBS tests, the initial contact of the shearing knife-edge is at one point where the stress is concentrated in a smaller area, resulting in premature failure [40]. Besides, SBS tests might generate undesired stress distribution pattern causing cohesive failures within the VC [41]. With the test method used in this study framework-veneer interface is loaded circumferentially, contacting a larger area and thus preventing early failures. This might also help to explain the high bond strength values obtained.

None of the test designs can exactly reflect clinical conditions and most have been performed on flat models of bilayered zirconia. In fact, FDPS have complex geometries and therefore bond strength tests that have a flat model design would not be sufficient to evaluate the bond strength of VC close to clinical conditions as these testing methods do not take the compressive forces that reinforces the circumferentially applied veneering ceramic into consideration. The test method proposed by Shell and Nielsen, allows applying the load only to the shearing surface for ultimate bond strength between the framework material and the VC [23]. Firing the VC circumferentially to the framework more accurately mimics the clinical situation when compared to flat model designs. It has also been stated that the high bond strengths that were obtained with this specimen geometry were due to compressive radial stresses [30]. Leone and Fairhurst reported ultimate bond strength values at the interface, as their specimens were

free from veneer ceramic at failure with this test method [42]. Other studies have also reported lower SBS of 23-41MPa [21], 29 MPa [7], 22-31MPa [12], and with MTBS between 24.2-31 MPa [6,15], 34-36MPa [41] for veneered zirconia all-ceramic systems compared to our study. During the phase transformation of the ceramic from the solidifying temperature to room temperature, molten ceramic surface cools faster than the slow cooling process interior, which stimulates residual compression stresses in the ceramic surface and innocuous tensile stresses within the ceramic material [23]. This phenomenon might explain the higher bond strength values obtained in this study compared to flat bond strength tests.

Many interacting variables such as compatibility of CTE, the zirconia surface conditioning, the liner application, the type of VC, framework-veneer bond quality and multiple firing of VC, being also a critical factor that may influence the tetragonal to monolithic phase transformation of zirconia and residual stresses, may affect the adhesion between these two ceramics [7,24,43]. Air-abrasion of zirconia surface before veneering results in high bond strength [1,7,24,37]. Accordingly, in this study, the specimens were air-abraded. It has been previously reported that excluding liner application did not have adverse effect on the bond strength [7,17,37,41]. Thus, no liner was applied to the zirconia in any of the test groups.

The delamination of VC from the zirconia framework is not directly related to the weak adhesion at the interface [20,37,38]. Residual stresses generated due to differential cooling should not be neglected. The mismatch of CTE between the framework and the veneer ceramic yields to increased interfacial failures as a result of increased tensile stresses at the interface and compressive stresses within the veneer ceramic [32]. It has been also reported that the mismatch of CTE causes high tensile pre-stress at the framework-veneer interface [6,43]. The perfectly matching CTE between the framework and the VC would result in minimum residual stress at the interface during slow cooling [23]. Hence, optimized CTE between the framework and VC could decrease delamination failures [2,7]. It was hypothesized that the cylindrical symmetry of the VC would not cause significant stress to the bond surface, and the matching CTE of framework and VC would avoid the thermal stresses that would have developed during cooling [23]. Shell and Nielsen stated that slight differences in CTEs had generated a large radial tensile stress at the framework-veneer interface; yet bond strengths had not been adversely affected [23].

Although there were slight differences between the CTEs of the VCs tested, the bond strength values did not reveal significant differences in most of the tested groups. An interesting finding of this study was that, CZ which had the least matching CTE ($9.2 \times 10^{-6} \text{K}^{-1}$) to that of the zirconia framework ($10.5 \times 10^{-6} \text{K}^{-1}$)

had the highest mean bond strength value (42.08 MPa) whereas IZ with the closest CTE ($9.6 \times 10^{-6} \text{K}^{-1}$) presented the lowest bond strength value (40.05 MPa). This might be a result of specimen geometry, radial compressive stresses, and the circumferential loading of the specimens. Interestingly however, Weibull distribution presented higher shape values (m) for IZ followed by MM both of which had the highest TEC being closest to that of the zirconia framework tested. Future studies should consider Weibull modulus in reporting data on adhesion of bilayered ceramics and focus on the correlation between such moduli and bond strength results using large number of specimens.

SEM evaluation revealed dominantly adhesive failures at the interface with all VC-zirconia combinations supporting that the specimens were loaded close to the framework at the interface to measure the ultimate bond strength. The mixed failure types and even the cohesive ones showed very thin layers of veneering ceramic as patches on the zirconia framework. This may also indicate that the adhesion of all VCs could be improved onto the zirconia, as the clinical problem of delamination remains to be a major concern in veneered zirconia FDPs.

Conclusion

From this study, the following could be concluded:

1. Shell Nielsen bond strength test used in this study subjecting the zirconia framework-veneering ceramic assemblies to circumferential shear forces did not show the effect of CTE mismatch between the veneering ceramics except those between CZ ($9.1 \times 10^{-6} \text{K}^{-1}$) and IZ ($9.6 \times 10^{-6} \text{K}^{-1}$).
2. The highest Weibull modulus was obtained with the veneering ceramic MM with TEC of $9.4 \times 10^{-6} \text{K}^{-1}$ indicating more reliable adhesion to the tested zirconia framework material.
3. Regardless of differences in TECs all zirconia framework-veneer-ceramic combinations presented predominantly adhesive failures after the bond test indicating an unfavourable failure type.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

References

1. Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure analysis of a selection of all-ceramic materials. Part II. Zirconia based dental ceramics. *Dent Mater* 2004;20:440-456.
2. Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systemic review. *J Prosthet Dent* 2007;98:389-404.
3. Denry I, Kelly Jr. State of art of zirconia for dental applications. *Dent Mater* 2008;24:299-307.
4. Ozyurt Z, Kazazoglu Z, Unal A. In vitro evaluation of shear bond strength of veneering ceramics to zirconia. *Dent Mater J* 2010;29:138-146.
5. Saito A, Komine F, Blatz MB, Matsumura H. A comparison of bond strength of layered veneering porcelains to zirconia and metal. *J Prosthet Dent* 2010;104:247-257.
6. Isgro G, Pallav P, van der Zel JM, Feilzer AJ. The influence of the veneering porcelain and different surface treatments on the biaxial flexural strength of a heat-pressed ceramic. *J Prosthet Dent* 2003;90:465-473.
7. Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Effect of loading method on the fracture mechanics of two layered all-ceramic restorative systems. *Dent Mater* 2007;23:952-959.
8. Roediger M, Gersdorff N, Huels A, Rinke S. Prospective evaluation of zirconia posterior fixed partial dentures: four-year clinical results. *Int J Prosthodont* 2010;23:141-148.
9. Raigrodski AJ, Hillstead MB, Meng GK, Chung KH. Survival and complications of zirconia-based fixed dental prostheses: a systematic review. *J Prosthet Dent* 2012;107:170-177.
10. Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, Mercante DE. The efficacy of posterior three-unit zirconium- oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. *J Prosthet Dent* 2006;96:237-244.
11. Ozkurt Z, Kazazoglu E. Clinical success of zirconia in dental applications. *J Prosthodont* 2010;19:64-68.
12. Al-Dohan HM, Yaman P, Dennison JB, Razzoog ME, Lang BR. Shear strength of core-veneer interface in bi-layered ceramics. *J Prosthet Dent* 2004;91:349-355.
13. Guazzato M, Proos K, Sara G, Swain MV. Strength, reliability, and mode of fracture of bilayered porcelain/core ceramics. *Int J Prosthodont* 2004;17:142-149.

14. Rocha EF, Anchieta RB, Freitas AC Jr, de Almeida EO, Cattaneo PM, Chang KO C. Mechanical behavior of ceramic veneer in zirconia-based restorations: a 3-dimensional finite element analysis using micro- computed tomography data. *J Prosthet Dent* 2011;105:14-20.
15. Tinschert J, Zvez D, Marx R, Anusavice KJ. Structural reliability of alumina-, feldspar-, leucite-, micra- and zirconia- based ceramics. *J Dentistry* 2000;28:529-535.
16. White SN, Miklus VG, McLaren EA, et al: Flexural strength of a layered zirconia and porcelain dental all-ceramic system. *J Prosthet Dent* 2005;94:125-131.
17. Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: Zirconia veneering ceramics. *Dent Mater* 2006;22:857-863.
18. Kern M. Controlled airborne-particle abrasion of zirconia ceramic restorations. *J Prosthet Dent* 2010;103:127-128.
19. Yang B, Barloi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. *Dent Mater* 2010;26:44-50.
20. Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann H. Fracture resistance of lithium disilicate-, alumina-, and zirconia-based three-unit fixed partial dentures: a laboratory study. *Int J Prosthodont* 2001;14:231-238.
21. Dundar M, Özcan M, Comlekoglu E, Gungor MA, Artunc C. Bond strengths of veneering ceramics to reinforced ceramic core materials. *Int J Prosthodont* 2005;18:71-72.
22. Fischer H, Marx R. Fracture toughness of dental ceramics: comparison of bending and indentation method. *Dent Mater* 2002;18:12-19.
23. Shell JS, Nielsen JP. Study of the bond between gold alloys and porcelain. *J Dent Res* 1962;41:1424-1437.
24. Ban S, Anusavice J. Influence of test method on the failure stress of brittle dental materials. *J Dent Res* 1990;60:1791-1799.
25. Özcan M. Fracture reasons in ceramic-fused-to-metal restorations. *J Oral Rehabil* 2003;30:265-269.
26. Comlekoglu ME, Dundar M, Özcan M, Gungor MA, Gokce B, Artunc C. Evaluation of bond strength of various margin ceramics to a zirconia ceramic. *J Dent* 2008;36:822-827.
27. Kotousov A, Kahler B, Swain M. Analysis of interfacial fracture in dental restorations. *Dent Mater* 2011;27:1094-1101.

28. Sundh A, Sjogren G. A comparison of fracture strength of yttrium-oxide–partially-stabilized zirconia ceramic crowns with varying core thickness, shapes and veneer ceramics. *J Oral Rehabil* 2004;31:682-688.
29. Guess PC, Kulis A, Witkowski S, Wolkewitz M, Zhang Y, Strub JR. Shear bond strengths between different zirconia cores and veneering ceramics and their susceptibility to thermocycling. *Dent Mater* 2008;24:1556-1567.
30. McLean, JW, and Sced IR. The Gold Alloy/Porcelain Bond, *Trans Brif Ceram Soc* 1973;5:229-233.
31. Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part 3: double veneer technique. *J Prosthodont* 2008;17:9-13.
32. Taskonak B, Mecholsky J, John J, Anusavice KJ. Residual stresses in bilayer dental ceramics. *Biomaterials* 2005;26:3235-3241.
33. Hermann I, Bhowmick S, Zhang Y, Lawn BR. Competing fracture modes in brittle materials subject to concentrated cyclic loading in liquid environments: trilayer structures. *J Mater Res* 2006;21:512-521.
34. Choi JE, Waddell JN, Swain MV. Pressed ceramics onto zirconia. Part 2: indentation fracture and influence of cooling rate on residual stresses. *Dent Mater* 2011;27:1111-1118.
35. Mainjot AK, Schajer GS, Vanheusden AJ, Sadoun MJ. Residual stress measurement in veneering ceramic by hole-drilling. *Dent Mater* 2011;27:439-444.
36. Goracci C, Sadek FT, Monticelli F, Cardoso PE, Ferrari M. Influence of substrate, shape, and thickness on microtensile specimens' structural integrity and their measured bond strengths. *Dent Mater* 2004;20:643-654.
37. Sundh A, Molin M, Sjogren G. Fracture resistance of yttrium oxide partially-stabilized zirconia all-ceramic bridges after veneering and mechanical fatigue testing. *Dent Mater* 2005;21:476–82.
38. Vult von Steyern P, Carlson P, Nilner K. All-ceramic fixed partial dentures designed according to the DC-Zirkon technique. A 2-year clinical study. *J Oral Rehabil* 2005;32:180-187.
39. Hadavi F, Hey JH, Ambrose ER, Louie PW, Shinkewski DJ. The effect of dentin primer on the shear bond strength between composite resin and enamel. *Oper Dent* 1993;18:61-65.
40. Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. *J Dent* 1989;17:61-1767.

41. Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. *Dent Mater* 2005;21:984-991.
42. Leone EF, Fairhurst CW. Bond strength and mechanical properties of dental porcelain enamels. *J Prosthet Dent* 1967;18:155-159.
43. Isgro G, Kleverlaan CJ, Wang H, Feilzer AJ. Thermal dimensional behavior of dental ceramics. *Biomaterials* 2004;25:2447-2453.

Veneering Ceramic Material	Manufacturer	Chemical Composition	Batch number	CTE (50-500 °C 10 ⁻⁶ K ⁻¹)
Vita VM9 (VZ)	Vita Zahnfabrik Rauter GmbH&Co KG, Säckingen, Germany	SiO ₂ , Al ₂ O ₃ , K ₂ O, Na ₂ O, TiO ₂ , CeO ₂ , ZrO ₂ , CaO, B ₂ O ₃ , BaO, SnO ₂ , Mg ⁺ , Fe ⁺ , P ⁺ , O ₂ .	15110	9 - 9.2
Cerabien ZR (CZ)	Noritake Kizai, Co. Ltd. Nagoya, Japan	SiO ₂ , Al ₂ O ₃ , Na ₂ O, CaO, K ₂ O, MgO, LiO ₂ , B ₂ O ₃ , pigments	MB30D923	9.1
Matchmaker Zr (MM)	Davis Schottlander & Davis Ltd. London, United Kingdom	SiO ₂ , Al ₂ O ₃ , B ₂ O ₃ , CaO, K ₂ O, pigments	S612	9.4
Ice Zirconia Ceramic (IZ)	ZirkonZahn GmbH, Bruneck, Germany	SiO ₂ , Al ₂ O ₃ , P ₂ O ₅ , K ₂ O, Na ₂ O, CaO, F, TiO ₂ , pigments	KEAA0501	9.6

Table 2. Brands, manufacturers, batch number, chemical composition and thermal expansion coefficient of veneering ceramics according to the manufacturer's data used in this study.

Material	Starting Temperature (°C)	Pre-drying time (min) closing time	Heating rate (°C/min)	Heating time (min)	Firing time (°C/min)	Vacuum holding time (min)	Slow cooling ending time (°C)
Vita VM9 (VZ)	500	6	55	7.27	910	6	Room temperature
Cerabien ZR (CZ)	600	5	45	7.2	930	1	Room temperature
Matchmaker Zr (MM)	450	6	45	8	810	1	Room temperature
Ice Zirconia Ceramic (IZ)	400	6	55	7.38	820	2	Room temperature

Table 3. Firing schedules of the veneering ceramics according to the manufacturers' instructions.

Veneering Ceramic Type	Number of Specimens (n)	Mean (SD) (MPa)	Failure Type ⁺	<i>m</i>	σ_0
VZ	10	41.77±4.92 ^{a,b}	7A, 1C, 2M	16.94	43.01
CZ	10	42.08±4.08 ^a	7A, 0C, 3M	18.3	43.33
MM	10	40.7±3.64 ^{a,b}	6A, 1C, 3M	20.16	41.75
IZ	10	40.05±5.78 ^b	5A, 1C, 4M	19.17	41.24

Table 4. Mean bond strength and standard deviation (SD) (MPa), failure types and Weibull distribution values including modulus (*m*), characteristic bond strength (σ_0), for each experimental group. *Different lower case superscripts indicate statistically significant difference (Tukey's test; $P<0.05$) ⁺Adhesive (A): Failure between the framework and the veneer ceramic without any remnants of the veneer ceramic; Cohesive (C): Failure within the veneering ceramic; Mixed (M): Combination of A and B. See Table 2 for groups abbreviations.

Figure Legends

Fig 1. Illustration of the test specimen preparation for Shell Nielsen bond strength test. c)

Zirconia rod (R:4mm, h:20mm). v) Veneering ceramic. m) Stainless steel mold. r) Metal rod (R:4mm, h:5mm) to create vertical clearance during testing.

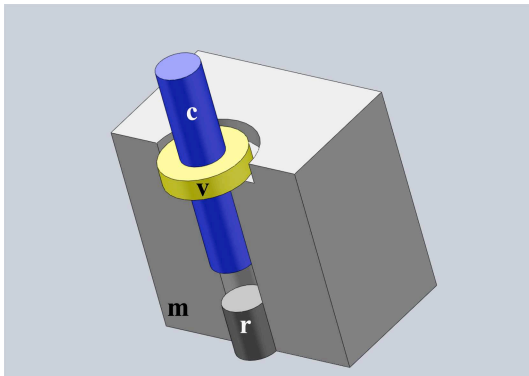


Fig 2. Specimens were seated in the mold used for specimen preparation and loaded from the top of the zirconia rods with a ball point (0.5 mm/min).

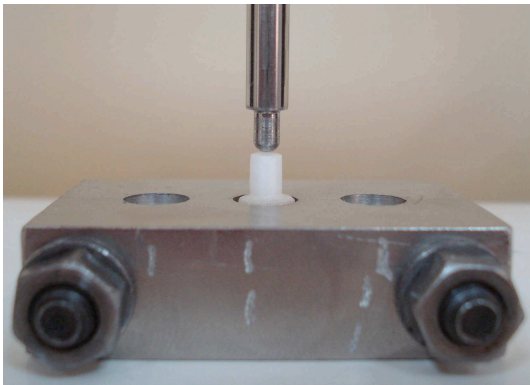


Fig 3. Fractured specimen.

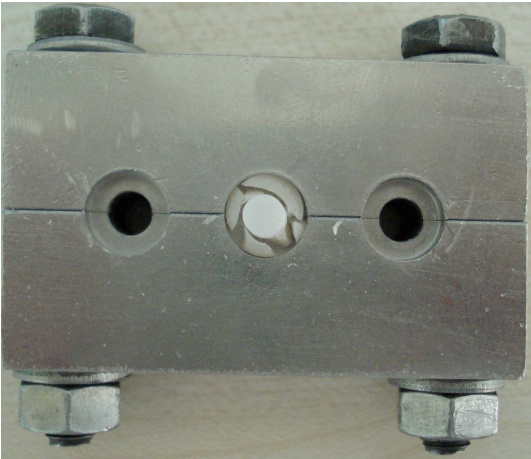


Fig 4. Weibull plot for the tested groups.

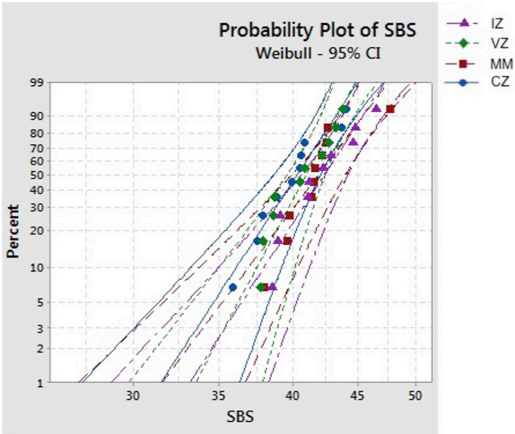


Fig 5. SEM images of the tested groups. **a)** SEM image (x35) of a representative from a CZ specimen. Good bond between core and the veneer without bubbles at the margins of. No voids at the margins but few within the veneering ceramic. **b)** Total delamination of the veneering ceramic from the zirconia core following Shell Nielsen bond strength test of a CZ specimen (x35). **c)** Zirconia core (VZ) free from the veneering ceramic after fracture indicating that ultimate bond strength at interface can be measured with Shell Nielsen test method (x35). **d)** Adhesive failure was prominent throughout the surface even though this failure type was classified as mixed (IZ) (x35). **e)** Higher magnification (X350) of the IZ specimen with a mixed failure type. Very thin layer of the veneering ceramic can be observed. **f)** Cohesive failure within the veneering ceramic (MM) at x35 magnification. Note the voids at the margins.

